



The influence of metal phase composition on microstructure and mechanical properties of Al₂O₃-Cu-Cr ceramic metal composites

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Abstract

Ceramic metal composites from the ternary Al₂O₃-Cu-Cr system were fabricated with the use of the slip casting method. Densities of the obtained composites with 15 vol.% of metal content and different percentage composition of Cu and Cr were examined by the Archimedes method. The samples' microstructures, observed by scanning electron microscope, were analysed with image analysis program in order to determine size distribution of the metallic areas in the structure. Phase composition of the sintered samples was examined by X-ray diffraction analysis. Vickers hardness of the samples was tested along with their fracture toughness with the use of the Vickers indentation technique. The conducted research revealed the correlation between the microstructure and mechanical properties of the obtained specimens. It has demonstrated the possibility of manufacturing composites from ternary system with the use of the slip casting method. It was revealed that the addition of the chromium particles to the metallic phase enhances hardness of the composites. Also, the metallic phase composition of the samples affects mechanism of the crack propagation in the material. Although the mechanical properties have been improved, it was found that the density of the samples was not satisfying and the need for further analysis has been identified.

Keywords: slip casting, ceramic-metal composite, Al₂O₃-Cu-Cr

I. Introduction

One of the challenges facing modern engineering today is the need for continuous improvement of the materials and their manufacturing technology in high-performance applications. Although, advanced ceramics with its remarkable mechanical properties can be considered as an appealing group of materials with a wide range of both current and future applications, its use is limited due to the low toughness and plasticity or high sensitivity to the presence of flaws. Taking that into consideration, the need for the new ceramic-based

materials development is justified [1–3]. Ceramic metal composites with their properties combining the advantages of both ceramics and metal seem to represent the promising direction for ceramic-based materials development [1–3].

Alumina, due to the well-known advantages like refractory, abrasive resistance, high hardness or chemical stability and accessibility can be used in many possible application fields. However, alike to other ceramic materials, alumina brittleness can be considered as the main limitation of its application [4,5]. Multiple researches have shown that introducing small amounts of metal particles into the alumina matrix can significantly improve the mechanical properties of the ma-

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terial and thus increase its application potential. Presence of different metallic phases such as nickel [6–8], aluminium [9], iron [10], molybdenum [11,12], tungsten [13], chromium [14–20] or copper [21–27] and their influence on the properties of the obtained ceramic metal composites with alumina matrix has been the area of scientific interest in recent years. In all of the researches, incorporation of the metal particles into the alumina matrix has been proven to enhance a fracture toughness of the obtained composites. Both copper and chromium have their unique properties, like high ductility, proper thermal and electrical conductivity in the case of Cu [21] and high corrosion, oxidation resistance and mechanical strength in the case of Cr [14]. Therefore, both of these systems: Al_2O_3 -Cu and Al_2O_3 -Cr are quite well represented in the specialist literature. The literature review on the Al_2O_3 -Cu system indicates that this type of ceramic-metal composites can be efficiently fabricated by means of various manufacturing techniques such as combination of uniaxial pressing and pressureless sintering [26], hot pressing [21–25] or slip casting [27]. Available research has proven that the addition of small amounts of copper into alumina ceramic matrix resulted in the improvement of the mechanical properties [21,26] and greater shock resistance of the composite material in comparison to the pure Al_2O_3 ceramics [23]. Scientific literature in the case of Al_2O_3 -Cr system indicates the possibility of use of the hot pressing [14,15], uniaxial pressing with pressureless sintering [14,18], combustion synthesis [19], mechanical and mechanical-thermal synthesis [17] or slip casting [20] as the efficient fabrication techniques. In all of the conducted studies, chromium addition to the alumina matrix caused enhancement of the mechanical properties of the material and its corrosion resistance. Although many studies have been carried out on different ceramic-metal systems, knowledge about composites with ceramic matrix incorporated with a metal phase consisting of more than one metal component still requires attention.

This work focused on the fabrication and characterization of ceramic metal composites from the ternary Al_2O_3 -Cu-Cr system. Composites with up to 15 vol.% of metal phase with respect to the total solid volume were prepared with the use of the slip casting method and pressureless sintering. The metal phase composition was a differentiating factor in the obtained composite samples. Selected physical and mechanical properties of the fabricated composite samples and its correlation with the microstructure and metal phase composition have been investigated.

II. Experimental

Composite synthesis was carried out with the use of following powders: α - Al_2O_3 A16SG (Almatis, USA) with average particle size $0.5\ \mu\text{m}$ and density $3.96\ \text{g/cm}^3$, Cu powder (Sigma Aldrich) with average particle size varied from 14 to $25\ \mu\text{m}$ and density

$8.94\ \text{g/cm}^3$ and Cr powder (Createc, Poland) with average particle size $> 50\ \mu\text{m}$. Characterization of the powders was performed on the basis of data provided by the producers. Alumina, due to its high hardness, satisfactory strength properties, high chemical and thermal shock resistance and low cost in comparison to other ceramic materials can be considered as one of the most widespread ceramic oxide materials in advanced ceramics manufacturing. The use of the Al_2O_3 A16SG powder was associated with its larger grain size than other commercially available alumina powders such as Al_2O_3 TM-DAR (Taimei Chemicals, Japan). The use of ceramic powder with micrometric particle size enables the prepared slurry to liquefy easily. Consequently, this may result in obtaining composite samples with homogeneous metal phase distribution in the ceramic matrix.

In the presented investigations, the composite samples were obtained with the use of the slip casting method in the multi-stage fabrication process. Manufacturing begins with weighing of the initial powders in defined proportions and mixing them with defloculant and solvent to produce a slurry. For the preparation of the slurry, distilled water was used as a solvent. Ammonium salt of polyelectrolyte, commercially known as DURAMAX D3005, was used as a dispersant in an amount of 1.5 wt.% with respect to the total weight of the solid phase. The produced slurry was mixed and degassed in the planetary centrifugal mixer for automatic slurry mixing and degassing Thinky ARE-250. The slurry was mixed for 8 min at the rate of 1000 rpm and then degassed for 2 min at the rate of 2000 rpm. Prepared slurry, in the next step, was cast into gypsum moulds. The use of a porous mould allowed the removal of a solvent from the prepared suspension as a result of the capillary action force which resulted in the green body. In the next step, green bodies were removed from the gypsum moulds and dried for 24 h at $40\ ^\circ\text{C}$ and sintered at $1400\ ^\circ\text{C}$ with 2 h dwell time in reducing atmosphere consisting of 20% H_2 and 80% N_2 . Despite the fact that composites fabricated from A16SG alumina should be sintered at $1550\ ^\circ\text{C}$ according to the literature data [28], the authors decided to attempt to sinter the produced composites at a lower temperature equal to $1400\ ^\circ\text{C}$. This selection of the sintering temperature in the research was determined by the low melting point of copper ($1084\ ^\circ\text{C}$) [29] as one of the metal phase components. The authors assumed that the use of a lower sintering temperature will prevent liquid metal from leaking during the sintering process.

Six samples with 50 vol.% of solid content and 15 vol.% of metal content with respect to the total solid volume were prepared. The metal phase consisted of different percentages amount of Cu and Cr in each sample, as it is seen in Table 1. Several methods were used to determine the properties and microstructure of the sintered composites. X-ray diffraction analysis was carried out to determine the phase composition of the sintered composites. The analysis was performed by

Table 1. Characteristics of the manufactured composite samples

Type of Al ₂ O ₃ - Cu-Cr samples	Solid content [vol.%]	Metal content [vol.%]	Cu content in metal phase [%]	Cu content in metal phase [%]
Series I	50	15	100	0
Series II	50	15	80	20
Series III	50	15	65	35
Series IV	50	15	50	50
Series V	50	15	35	65
Series VI	50	15	20	80

Rigaku Miniflex II X-ray diffractometer with Cu K α ($\lambda = 1.54178 \text{ \AA}$, 30 kV, 15 mA) radiation in the 2θ range from 20° to 100° at a step size 0.02° with 5 s counting time.

The Archimedes method was used to measure apparent and relative density or open porosity. The real density of the sintered samples was measured by the Accu Pyc II 1340 helium pycnometer. The measurements were carried out in the two stages: 10 purges and 600 measurements cycles with 0.13 MPa fill pressure for both stages. Microscopic examinations were carried out in order to visualize the morphology of the metallic powders and microstructure of the cross-sections of the sintered samples. Observations of the metallic powders were performed with the scanning electron microscope JSM-6610 with a secondary electron detector (SE). Also, the microstructure of the fabricated composites was investigated using scanning electron microscope Hitachi TM-1000 equipped with a backscattered electron detector (BSE). The samples were cut using a precision diamond saw and then mounted in resin, ground with abrasive paper in the range of 80–4000 gradations and polished using diamond pastes (3 and 1 μm gradation). Image analysis with the use of the Micrometer computer software was performed [30] on the SEM micrographs of the prepared cross-sections of the samples. On the basis of the average equivalent diameter of the metallic areas in the microstructure, determined by

the software, the percentage share of different size fractions of the metallic particles was determined. The analysis for all samples was performed for ten microstructure micrographs from different areas of the sample.

Vickers hardness was measured with the use of a Vickers hardness tester HPO-250 under the load of 196 N and holding time of 10 s. In the experiment for each specimen, 12 hardness measurements and 10 crack length measurements were made. The obtained results represented the average value of the hardness based on ten indentation measurements. Photographs of the indentations and the measurements of the diagonals were captured with the SEM Hitach TM-1000. The K_{IC} values for each sample were determined by the Vickers indentation crack length method. In this method fracture toughness was examined based on the length measurements of the cracks propagated from the corners of the Vickers indentation. The Niihara equation was used to calculate K_{IC} values for each sample [31]:

$$K_{IC} = 0.018 \cdot HV^{0.6} \cdot E^{0.4} \cdot 0.5(2a) \cdot l^{-0.5} \quad (1)$$

for $0.25 < l/a < 2.5$. In this equation HV is Vickers hardness, E is Young's modulus, a is half of indentation diagonal length and l is crack length.

The presented research has a preliminary character and the gained results can be treated as a prerequisite for acquiring the new knowledge in the ternary ceramic metal system area and its properties.

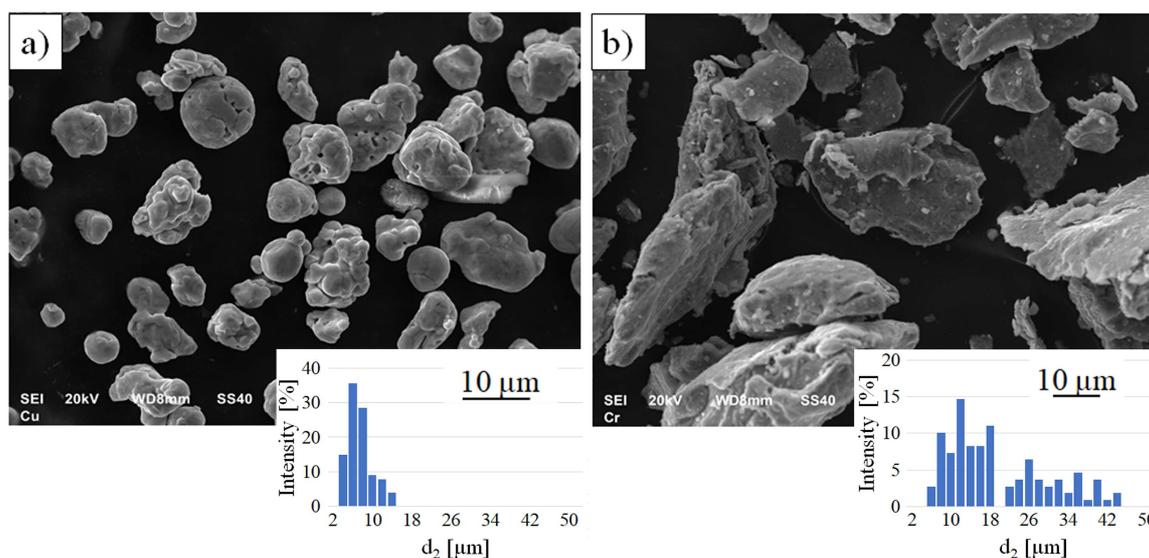


Figure 1. SEM micrographs and particle size distribution histograms for initial metal powders: a) Cu powder, b) Cr powder

III. Results and discussion

To characterize powders microscopic observations on the scanning electron microscope and particle size distribution analysis of the initial metal powders were performed. SEM micrographs analysis (Fig. 1) showed significant diversification in the morphology of the metal powders applied in the study. The copper powder was characterized by the spheroidal shape of particles, while chromium powder had irregular morphology. Image analysis of the SEM micrographs of the metal powders was also performed to determine the average particle size of both metal particles (Fig. 1). It showed that the copper powder used in the research can be characterized by particle size in the range from 4 to 13 μm with an average particle size equal to 6 μm . In the case of the chromium particles, it was observed that it is characterized by a greater variety of particle sizes, which were in the range from 8 to 50 μm with a small fraction of the particles above 50 μm . On the basis of calculations made from the micrographs (an example is shown in Fig. 1b), it was found that the average grain size for the chromium powder is equal to 18.8 μm .

X-ray diffraction patterns of the sintered specimens are shown in Fig. 2 and confirm the presence of alumina and copper in the samples without chromium, while in the specimens with the different amounts of chromium addition the presence of alumina, copper and chromium were revealed during the analysis. It can be observed that regardless of the metal phase composition, no new phases were present in the analysed composite specimens during the measurement. Additionally, the intensity of the peaks representing metallic phase components varied with the metal phase composition. The obtained results revealed that the use of a reducing atmosphere during the sintering process prevents metal phase oxidation. This results in the three-phase structure in the obtained composite specimens.

The selected physical properties of the analysed composite samples were presented in Table 2. Analysis of the obtained results based on Archimedes method indicated that the lowest relative density values characterized the samples with the predominance of chromium in the metallic phase. The lowest relative density was measured for the Series IV with equal amount of copper and chromium in the metallic phase and it was equal to $76.43 \pm 1.06\%$. The same sample was also characterized by the highest open porosity value among all of the examined specimens, which was equal to $21.16 \pm 0.79\%$. The highest relative density characterized the sintered sample from the Series II with 20% of chromium in the metallic phase. The important information from pycnometer measurement is the density of sintered specimens because it helps to determine how much metal may have flowed from the composites during the sintering process. Accordingly, in the next step, the real density of the composites was determined. It was found that the real density measured for the obtained samples is

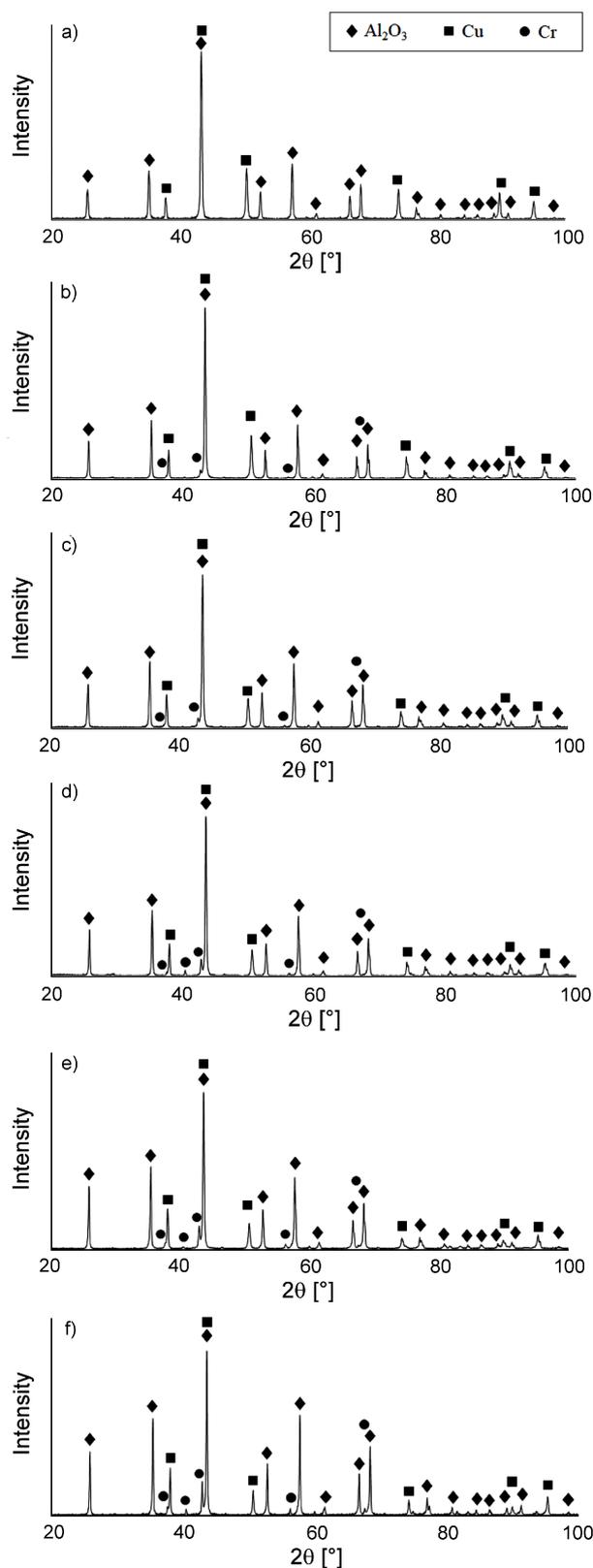


Figure 2. XRD patterns of composites: a) Series I: 0% Cr, 100% Cu, b) Series II: 20% Cr, 80% Cu, c) Series III: 35% Cr, 65% Cu, d) Series IV: 50% Cr, 50% Cu, e) Series V: 65% Cr, 35% Cu, f) Series VI: 80% Cr, 20% Cu

Table 2. Densities of the obtained composite samples in dependence of the amount of chromium addition in metal phase

Sample Cr content [%]	Series I 0%	Series II 20%	Series III 35%	Series IV 50%	Series V 65%	Series VI 80%
Theoretical density* [g/cm ³]	4.707	4.653	4.617	4.572	4.527	4.491
Apparent density** [g/cm ³]	3.88 ± 0.06	3.85 ± 0.06	3.76 ± 0.05	3.49 ± 0.05	3.52 ± 0.04	3.59 ± 0.05
Relative density** [%]	82.36 ± 1.25	82.74 ± 1.22	81.33 ± 1.17	76.43 ± 1.06	77.72 ± 0.94	79.82 ± 1.17
Open porosity*** [%]	16.55 ± 0.83	14.15 ± 1.21	14.54 ± 1.09	21.16 ± 0.79	17.36 ± 0.51	17.29 ± 1.53

*Calculated using rule of mixtures, **Measured by Archimedes method, ***Measured by pycnometer method

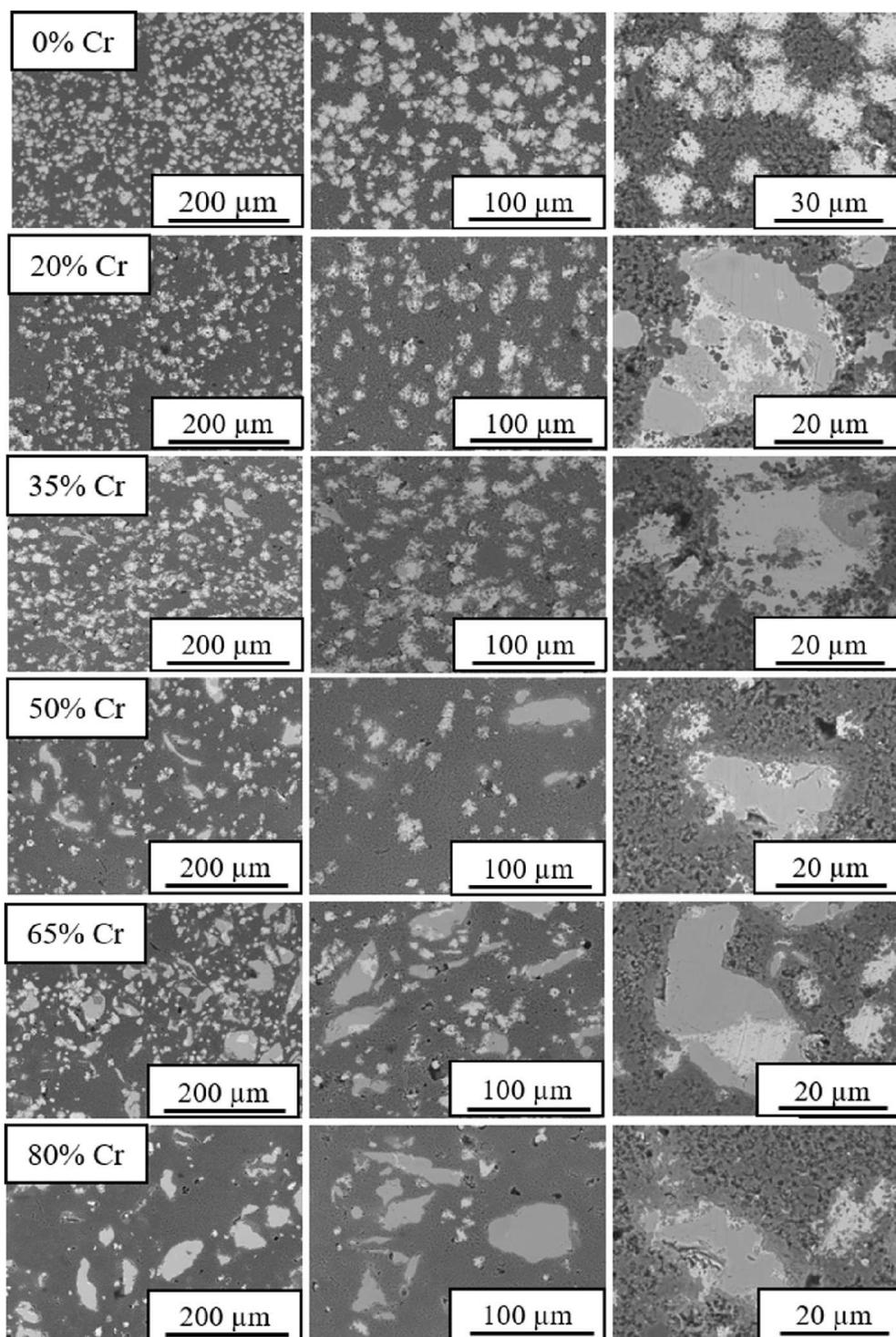


Figure 3. Microstructure of composites with different chromium content in metal phase

lower than theoretical density for all examined samples. This indicates the density lowering during the sintering process. Difficulties in sample densification are related to the observed outflow of liquid copper during the sintering process. In each sample approximately 2–3% of copper loss after sintering was observed. Unfortunately, this means that the use of a lower temperature does not prevent leakage of liquid metal during sintering. Therefore, it can be concluded that the obtained densities are not satisfying and have to be optimized in order to accomplish improved mechanical properties such as fracture toughness of fabricated samples. It should take into account that the measurement results of density, especially in these types of composites, are heavily determined by the distribution of porosity in the specimens.

Macroscopic observations, performed on the sintered specimens (Fig. 3), confirm that there are no visible cracks on the surface of the manufactured samples. The observation demonstrated that dark areas in the microstructure stand for the Al_2O_3 matrix while white and grey particles correspond to copper and chromium respectively. It was found that the microstructure of the composites strongly depends on the metal phase composition. Based on the presented results it can be concluded that in the samples where the metal phase consists entirely or predominantly of copper, the presence of numerous metallic particles of irregular shape has been observed. Whereas, with the changing of copper and chromium contents in the metallic phase, larger metallic particles with the morphology similar to that of the initial chromium particles were observed in the microstructure. As the sintering temperature for the composite samples was higher than the copper melting point, it was assumed that the copper particles were present in the liquid state during the sintering process

which resulted in the morphology change of the copper particles in the sintered microstructure. The presence of copper was also observed between chromium particles in the observed sample microstructures. The conducted observations also revealed presence of the high porosity in the obtained specimens. Reduced sintering temperature, which was meant to limit Cu melting, also can cause remained porosity observed in the structure of the sintered samples.

The results of the SEM micrograph image analysis are presented in Fig. 4. They indicate that along with the increasing amount of the chromium in the metal phase, the percentage amount of the larger particles in the microstructure also increases. In the case of the samples with only copper in the metal phase, the main share in the microstructure was observed for particles in the range from 8 to 10 μm . Small additions of chromium to the metal phase result in the increasing amount of the metallic particles below 8 μm in the microstructure. Along with the increasing amount of chromium in the metallic phase the percentage of larger particles above 20 μm increases. In all samples with more than 50% of chromium, a fraction of the particles higher than 50 μm was also observed. It can be assumed that a small addition of chromium to the metal phase causes fragmentation of the metal particles in comparison to the reference Al_2O_3 -Cu sample. However, the amount of larger chromium particles in the metal phase leads to the increase of percentage of larger metallic areas with d_2 value above 25 μm in the structure of the obtained composites.

The Vickers hardness results, presented in Fig. 5, revealed the dependence between the hardness value and metal phase composition of the analysed specimens. It was proven that even small addition of chromium to

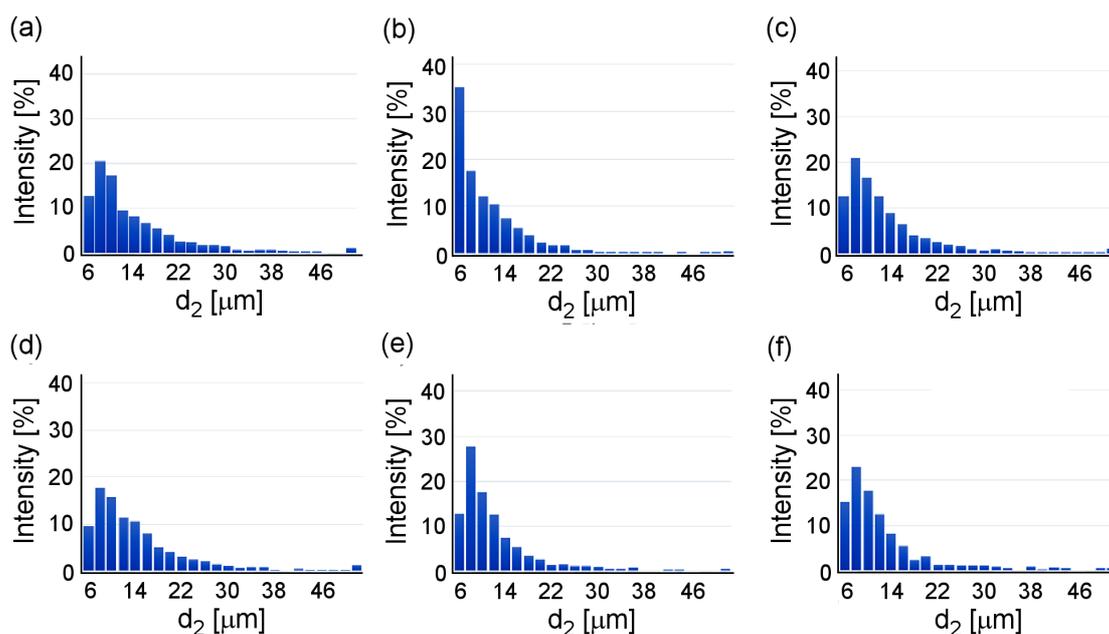


Figure 4. Image microstructure analysis results: a) Series I: 0% Cr, 100% Cu b) Series II: 20% Cr, 80% Cu, c) Series III: 35% Cr, 65% Cu, d) Series IV: 50% Cr, 50% Cu, e) Series V: 65% Cr, 35% Cu, f) Series VI: 80% Cr, 20% Cu

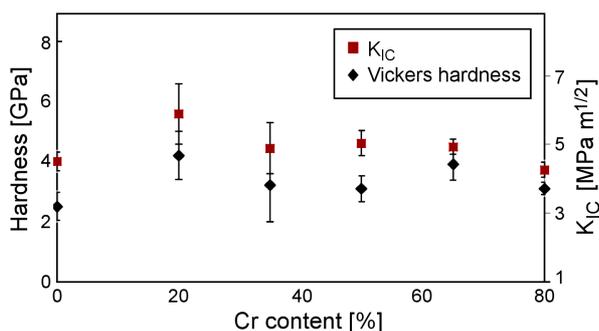


Figure 5. Vickers hardness and fracture toughness of the obtained specimens in dependence of Cr content in metal phase

the metal phase of the $\text{Al}_2\text{O}_3\text{-Cu-Cr}$ composites results in Vickers hardness values enhancement for the sintered specimens. The composite from the Series VI fabricated with metal phase consisted of 80% amount of chromium and 20% amount of copper was distinguished by the highest measured Vickers hardness value, equal to 4.2 ± 0.8 GPa. The obtained outcomes indicate that the lowest Vickers hardness (2.5 ± 0.46 GPa) characterized the Series I with metal phase consisted only of copper, without any chromium addition. The results for the rest of ternary system specimens (the Series II – Series V) were similar, in the range from 3.1 to 3.9 GPa.

Similar correlation can be observed in the case of fracture toughness. Analysis of the obtained results shows that the highest fracture toughness characterizes

the composite specimens from the Series II with 20% chromium and 80% copper in the metal phase. The K_{IC} value for this samples calculated with the Niihara equation was equal to 5.88 ± 0.88 $\text{MPa}\cdot\text{m}^{0.5}$. For comparison, K_{IC} value calculated with the use of same equation for the composite from the Series I without chromium in the metal phase was equal to 4.5 ± 0.47 $\text{MPa}\cdot\text{m}^{0.5}$. The reinforcement is likely a result of ceramic-metal bonding decohesion and, especially for the bigger metal inclusions, plastic deformation. The presence of the metallic particles in the composite forces the propagating crack to bypass or stretch them. The dissipation of crack energy, both as a result of the plastic deformation and the necessity of changing the crack propagation path, slows down the crack and thus enhancing fracture toughness of the material. The obtained hardness and fracture toughness results are inconsistent with the values reported by Ji *et al.* [32] where addition of chromium to the metal phase effected with composite hardness deterioration but significant fracture toughness improvement.

Vickers indentation analysis allowed to observe influence of metal phase presence on the crack propagation in the composite samples (Fig. 6). Several mechanisms of crack propagation inhibition were observed during the analysis, i.e. crack deflection on the metallic particles or crack bridging. Due to the different characteristics of the metal components in the metal phase it was observed that crack propagation mechanism is correlated with the metal phase composition. The Series with predominance of ductile copper in the metal phase (the

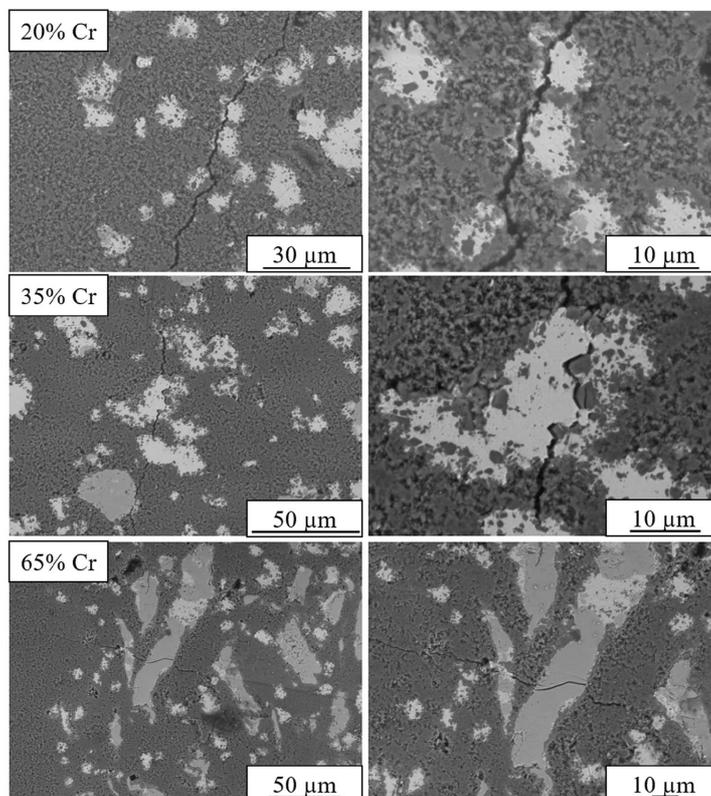


Figure 6. Illustrative SEM micrographs of the crack propagation inhibition mechanism in samples with different metal phase composition

Series III with 35% of Cr and 50% of Cr) were distinguished by crack deflection as the main mechanism for crack propagation inhibition. The increasing amount of harder chromium particles in the microstructure had an effect of cracks propagation through the metal particles which resulted in increasing amount of crack fragmentation mechanism as a main crack propagation inhibition mechanism in the observed samples (65% of Cr). Both mechanisms were previously observed in the literature. Ji *et al.* [32] indicated crack deflection as an accompanying mechanism for fracture toughness in material with weak ceramic-metal bonding. Particle crack propagation was also observed in the case of large chromium particles.

IV. Conclusions

The conducted experiments showed that Al₂O₃-Cu-Cr composites have been successfully fabricated with the use of the slip casting method. These investigations reveal that through the selection of appropriate amounts of the metal components, it is possible to design the microstructure and optimize the selected properties of the composite in the Al₂O₃-Cu-Cr system. The metallic phase composition has significant influence on the manufactured composites revealed its influence on the wide range of properties. The addition of chromium to the metal phase of the composite samples enhances hardness and fracture toughness in comparison to the Al₂O₃-Cu composites fabricated under the same conditions. It has been shown that strengthening in the analysed composites can be considered as a result of different crack propagation inhibition mechanisms on the metal particles distributed in the structure. The experiments presented in the manuscript are preliminary studies. The work on this problem is in progress and new results will be published in succeeding paper.

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